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Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART II PRELIMINARY EXAMINATION OF PERFUMERY MATERIALS AND SAMPLES

(Second Revision)

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PART II PRELIMINARY EXAMINATION OF PERFUMERY MATERIALS AND SAMPLES

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Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART II PRELIMINARY EXAMINATION OF PERFUMERY MATERIALS AND SAMPLES

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Part II) (Second Revision) was adopted by the Indian Standards Institution on 25 January 1980, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1952 entitled as ' Methods of test for essential oils ' and subsequently revised in 1968 as ' Methods of sampling and test for natural and synthetic perfumery materials '. Taking cognizance of the need to incorporate instrumental methods of analysis now in vogue and to facilitate inclusion of additional test methods or changes in the existing test methods and also to align these test methods with the corresponding ISO Standards, the Committee concerned decided to revise and split the standard and publish individual test methods as separate parts of the original standard.

0.2.1 In order to facilitate proper referencing of the method of test given in this revised part of IS : 326, Amendment No. 1 is being issued separately to the relevant clause of IS : 326-1968* which would no longer be in force with the publication of this revision. The test method as given in this revised part will supersede the test method as given under **4** of IS : 326-1968*.

0.3 In the preparation of this standard, assistance has been derived from the following publication:

BS 2073 : 1976 Methods of test for essential oils. British Standards Institution.

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*Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part II) prescribes the special tests for the detection of common impurities and adulterants in natural and synthetic perfumery materials.

2. PRELIMINARY EXAMINATION

2.0 General — The original containers selected for sampling and the test samples drawn, as the case may be shall be opened and the condition of the contents noted immediately and reported, particular attention being given to the following.

2.1 Colour

2.2 Clarity

2.3 By-Note — Report if the material passes a by-note.

NOTE — For detailed olfactory assessment of natural and synthetic perfumery materials, refer to IS : 2284-1963†. This method is based on a comparison of a given material with its corresponding standard sample.

2.4 Determination of Content of Alcohol Insoluble solid Impurities

2.4.0 *Outline of the Method* — Extraction of the perfumery materials is done with ethanol and weighing of any residue after filtration.

2.4.1 *Reagent*

2.4.1.1 *Ethanol* — 95 percent (*v/v*).

2.4.2 *Apparatus*

2.4.2.1 *Hot-air-oven* — capable of being maintained at 105 ± 1°C.

2.4.3 *Procedure* — Weigh accurately about 20 g of the perfumery material into a conical flask. Add about 200 ml of ethanol, mix the contents of the flask thoroughly and filter through a sintered glass crucible (porosity 4 is suitable) which has previously been dried in the oven at 105°C and weighed. Wash any insoluble matter remaining in the flask into the

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*Rules for rounding off numerical values (*revised*).

†Method for olfactory assessment of natural and synthetic perfumery materials.

crucible, suck dry and then dry the crucible and its contents in the oven at 105°C until the loss of mass on drying in the oven for an additional 30 minutes does not exceed 1 mg.

2.4.4 *Calculation*

Content of alcohol-insoluble solid impurities,

$$\text{percent by mass} = \frac{100\ (m_3 - m_2)}{m_1}$$

where

m_1 = mass in g of the sample of perfumery material taken,
m_2 = mass in g of the empty sintered glass crucible, and
m_3 = mass in g of the sintered glass crucible and residue.

2.5 Determination of Water Content

2.5.0 *Outline of the Method* — Distillation of the perfumery materials is done with a water-immiscible solvent.

2.5.1 *Reagent*

2.5.1.1 *Heptane* — clear and free from visible water, or toluene.

2.5.2 *Apparatus*

2.5.2.1 *Dean and stark apparatus* — With a distillation vessel of 500 ml capacity and a receiver of either 2 ml or 7·5 ml capacity. Clean the apparatus thoroughly with a mixture of potassium dichromate and concentrated sulphuric acid, rinse with clean water and dry before use.

2.5.2.2 *Electric heating mantle or oil-bath*

2.5.3 *Procedure* — Weigh 20 ± 0·5 g of the perfumery material into the distillation vessel. Half fill the vessel with solvent and add a few dry porcelain chips to regulate boiling. Assemble the apparatus. Maintain the contents of the flask at the boil using the heating mantle or oil-bath until the level of the water layer in the receiver remains constant for 30 minutes. Cool the apparatus to room temperature and detach any globules of water from the condenser wall by means of the spray tube. Read the volume of water in the receiver.

2.5.4 *Calculation*

$$\text{Content of water, percent by mass} = \frac{V \times 100\, d}{m}$$

where

V = volume in ml of water collected in the receiver,

m = mass in g of perfumery material taken, and

d = density of water at the air temperature (0·997 1 g/ml at 27°C).

2.6 Preparation of the Material for Physico-Chemical Analysis

2.6.1 Remove as far as possible any visible water present in the sample by decanting, then add about 10 percent by mass of neutral, freshly ignited and powdered magnesium sulphate and shake the mixture vigorously from time to time during a period of 2 hours. Filter through paper and store if necessary in clean, dry, airtight, opaque, non-absorbent containers, preferably of glass or of metal on which the sample has no action. Fill the sample containers leaving a little air space to allow for expansion and immediately seal after filling. Use only new good quality corks or glass stoppers or screw-lids in the case of glass containers and new good quality corks or sore stoppers in the case of metal containers. To prevent contact with the sample, aluminium foil or other suitable material may be wrapped round corks. Protect samples from heat.

2.6.2 In the case of perfumery materials which are solid or partly solid at room temperature (oils of rose and guaiac wood) warm the material just sufficiently to liquefy it and proceed as above, maintaining the temperature so that it remains liquid throughout.

2.6.3 The colour of some dark coloured perfumery materials may be lightened by shaking them intermittently for 10 minutes with 1 percent (m/m) of tartaric or citric acid. Filter and proceed as above.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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